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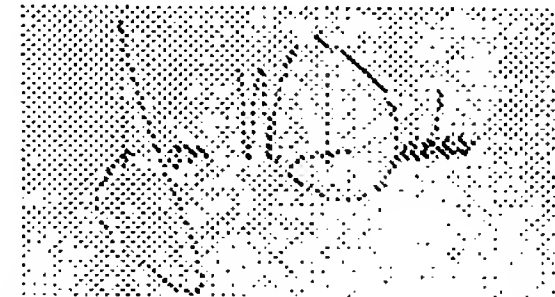
THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE.

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Certified by



Jon W Dudas

Acting Under Secretary of Commerce
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PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION under 37 CFR 1.53 (b)(2)

Docket Number	81,636	Type a plus sign (+) inside this box →
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TITLE OF THE INVENTION (280 characters max)

LOW ODOR BACK END CURED CATALYST FOR PIR AND PUR FOAMS AND ELASTOMERS

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ENCLOSED APPLICATION PARTS (check all that apply)

☒ Specification Number of Pages 7 ☐ Small Entity Statement
☐ Drawing(s) Number of Sheets 0 ☒ Other (specify) Postcard

METHOD OF PAYMENT (check one)

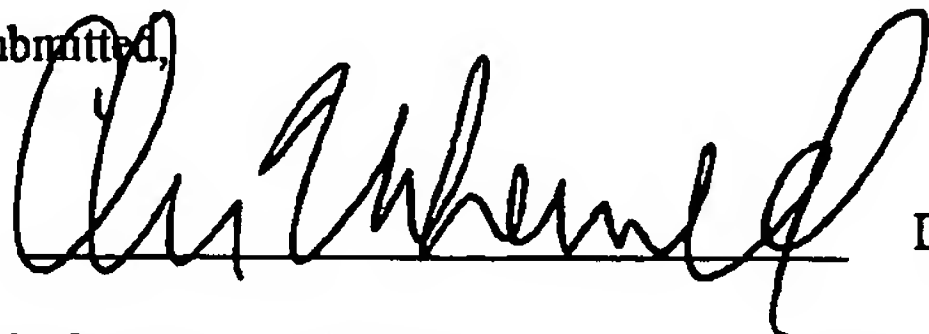
<input type="checkbox"/> A check or money order is enclosed to cover the Provisional filing fees	PROVISIONAL FILING FEE AMOUNT	\$ 160.00
<input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge the filing fee to Deposit Account Number: <u>08-3442</u>		

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

☒ No.
☐ Yes, the name of the U.S. Government agency and the Government contract number are:

Respectfully submitted,

SIGNATURE



Date

7/25/03

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I hereby certify that this correspondence is being deposited with the U.S. Postal Service Express Mail Service No. EU103960415US addressed to Commissioner for Patents, P. O. Box 1450, Alexandria, VA 22313-1450 on July 25, 2003.


Martha Victory

☐ Additional inventors are being named on separately numbered sheets attached hereto.

PROVISIONAL APPLICATION FILING ONLY

Low Odor Back-end Cured Catalyst for PIR and PUR Foams and Elastomers**Technical Field**

The present invention relates to catalyst compositions. More particularly it relates to aqueous quaternary ammonium catalysts useful in producing isocyanate-derived foams.

Background Information

US Patent 3,980,594 describes the trimerization of aromatic isocyanates catalyzed by certain ammonium salts. Quaternary ammonium salts or inorganic and organic oxygen acids having pK values for at least one of the dissociable hydrogen atoms equal to or greater than 2.0 in aqueous solution are employed as extremely efficient catalysts for trimerization of organic isocyanates, particularly aromatic isocyanates, to isocyanurates and for urethane formation.

US Patent 4,521,545 describes the latent catalyst made from an amine and a alkylating ester of an acid of phosphorous.

US Patent 4,582,861 describes the use and synthesis of N-hydroxyalkyl quaternary ammonium carbonate salt.

US Patent 4,785,025 and 4,904,629 describe the use and synthesis of salts based on TEDA.

Quaternary ammonium salts have been known to function as delayed action catalysts, improving on the cure of the final foams. Typically, they are made from a starting material of trimethyl amine, which has a very strong amine odor associated with

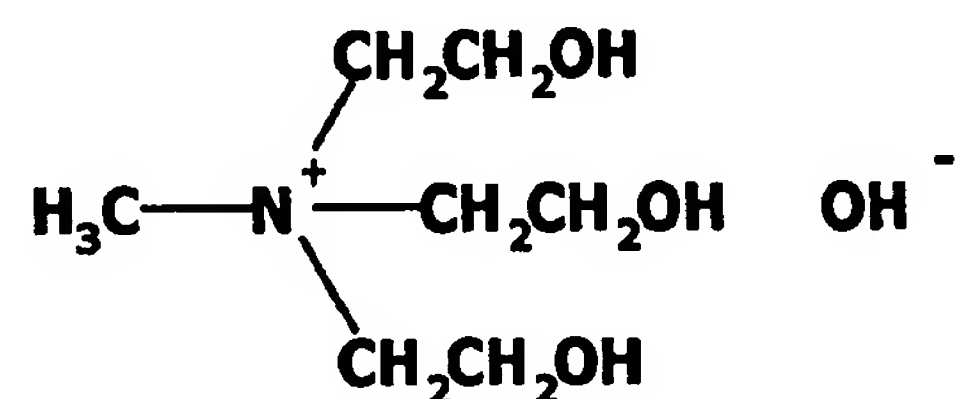
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it. These salts all have the anion derived from various acids. For a catalyst according to the present invention, the material is produced from methyldiethanol amine and hydroxide anion. Thus, it is possible to take this product and react it with various acids to make similar ammonium salts of various acids, but low in odor.

Detailed Description

The present invention is directed at using a particular quaternary ammonium salt as part of the catalyst package used in the curing of urethane foams and elastomers to improve isocyanate conversion and to decrease the time it takes to finish the reaction of the isocyanate with the reactive components while preventing a strong ammonia-like odor in the foam.

The quaternary ammonium salt useful as a co-catalyst according to the present invention contains the tris-(hydroxyethylmethyl)ammonium cation, and also contains an anion for charge neutrality. The structure of tris-(hydroxyethylmethyl)ammonium hydroxide ("JEFFCAT(R)TR-62") is as shown below:



The foams which are to be formed using a catalyst according to the present invention can be rigid PIR or PUR foams, flexible foams, and/or elastomeric foams. A catalyst according to the present invention is sometimes referred to as a "back end cure" catalyst by those skilled in the art, since it improves on the curing rate and conversion of the material.

According to a process of the invention, a catalyst according to the invention is used in conjunction or combination with other amine catalysts known to those skilled in

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the art to improve on the cure time and shorten the de-mold time of various polyurethane and polyisocyanurate foam and elastomer systems.

One application area where the catalyst of the present invention can be used quickly is in the end use manufacture of boardstock foam. In such an employment, the catalyst according to the present invention assists in converting the excess isocyanate to trimer materials in the foam.

COMPONENTS	PARTS BY WEIGHT	control
STEPHAN PS-2352	100	
Goldschmidt B-84PI	2.0	
141B	16	
Water	1.0	
RUBINATE®M(3.96 index)	283.8	
Actual weights used, grams		
B-component, g	31.8	31.8
A-component, g	75.7	75.7
Pelron 9540A. g	1.3	1.3
JEFFCAT(R)TR-62, g	0.5	
Cream time, seconds	11	12
Rise time, sec	62	59
Tack free time, sec	34	26
Density, g/ml	2.67	3.12

Example I

Unexpected or surprising results or advantages of the invention include the fact that conversion of the isocyanate at lower temperatures will occur when the appropriate acid block catalyst is used to catalyze the reaction of the isocyanate with the hydroxyl containing materials. The conversion at lower temperatures can shorten the time required for the material to be in a press. This will lead to faster cycle times with improve

productivity. Lower density foam was obtained when we used JEFFCAT(R)TR-62 in the formulations.

The following examples show the effect of JEFFCAT(R)TR-62 vs. JEFFCAT®TMR-2. In these examples, different levels of water were added to give the same amount of blowing as in the examples prepared using JEFFCAT®TMR-2. Again, we see lower density foams.

Formulation	I	II	III	IV	V	VI
Aromatic polyester polyol	100	100	100	100	100	100
PELCAT® 9540a	4.2	4.2	4.2	4.2	4.2	4.2
Surfactant	2.0	2.0	2.0	2.0	2.0	2.0
JEFFCAT(R)TR-62	0.25	0.5	0.75	-	-	-
JEFFCAT® TMR-2	-	-	-	0.25	0.50	0.75
Water	0.375	0.25	0.125	0.5	0.5	0.5
Pentane	21	21	21	21	21	21
RUBINATE® 1850	300	300	300	300	300	300
Rise profile						
Cream time, sec	9	8	8	9	9	9
Top of Cup, sec	23	18	19	21	21	19
Gel time, sec	34	26	25	30	30	28
Tack free time, sec	44	39	38	40	38	40
Rise time, sec	77	66	64	71	73	66
Firm time, sec	97	94	83	94	94	84
Free rise density, g/ml	1.53	1.57	1.65	1.69	1.70	1.74

Alternative, or other reactants useful with JEFFCAT(R)TR-62 include: Polyester polyols, polyether polyols, Mannich polyols, sucrose polyols, surfactants, either organic (carbon) or silicon based, potassium salts of various acids, other amine catalysts, blowing agents such as hydrocarbon, carbon dioxide, 141b, 245FA.

What is claimed is:

- 1) In a process for producing an elastomer, or a PIR or PUR foam product by reaction of an organic isocyanate and a polyol in the presence of a tertiary amine catalyst, wherein the improvement comprises conducting the reaction in the further presence of an effective catalytic amount of tris-(hydroxyethylmethyl)ammonium cation.
- 2) A process according to claim 1 wherein the charge counterbalance for said tris-(hydroxyethylmethyl)ammonium cation is provided by the presence of an ion selected from the group consisting of: hydroxide ion, carbonate ion, bi-carbonate ion, chloride ion, bromide ion, or a C₁-C₂₀ carboxylic acid anion.
- 3) A process according to claim 1 wherein said process is conducted in the presence of any amount of water between 0.001 % and about 60 % by weight based on the total weight of said polyol present.
- 4) A process according to claim 1 wherein said tertiary amine is selected from the group consisting of: JEFFCAT® ZF-10, JEFFCAT® ZF-20, JEFFCAT® ZR-40, and DMCHA, JEFFCAT® TD-33A, JEFFCAT® DMEA, JEFFCAT® ZR-70, JEFFCAT® DPA, JEFFCAT® ZR-50, JEFFCAT® Z-130, JEFFCAT® TR-90, JEFFCAT® TR-52, imidazoles, and PMDETA.
- 5) A process according to any of claims 1-4 which is conducted in the further presence of potassium ion.

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6) A process according to any of claims 1-5 which is conducted in the further presence of one or more materials known to those skilled in the art falling within the classes of: surfactants, flame retardants, and blowing agents when used in the production of polyurethane elastomers or polyurethane foams.

7) An aqueous solution comprising tris-(hydroxyethylmethyl)ammonium hydroxide, water, and a tertiary amine.

8) An aqueous solution according to claim 7 wherein said tertiary amine is selected from the group consisting of: JEFFCAT® ZF-10, JEFFCAT® ZF-20, JEFFCAT® ZR-40, and DMCHA, JEFFCAT® TD-33A, JEFFCAT® DMEA, JEFFCAT® ZR-70, JEFFCAT® DPA, JEFFCAT® ZR-50, JEFFCAT® Z-130, JEFFCAT® TR-90, JEFFCAT® TR-52, imidazoles, and PMDETA.

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